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Sustainable gypsum composites reinforced with basalt technogenic nanofiber

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Abstract. Sustainable composites based on gypsum man-made stone are produced using a technology that excludes the firing stage. It meets the requirements for resource and energy conservation, does not harm the environment and can be used in the production of a number of biopositive building materials. The use of pure dihydrate gypsum from gypsum mold waste in the composition of sustainable gypsum composites predetermines the expansion of the scope of application of materials and products based on it. These wastes are characterized by stable physicochemical and mechanical properties. However, the features of the mineralogical composition require high costs for their use in the production of fired gypsum binders using classical technologies. Binders based on them have low strength and other physical indicators. Application without firing technology allows for the maximum use of all the unique properties of gypsum – creation of a comfortable environment, high resistance in fire conditions, good insulating characteristics, etc. By introducing highly dispersed basalt dust particles into the composition of stable gypsum composites based on dihydrate gypsum, gypsum stone is reinforced and compacted at the micro- and nanoscale levels. This is facilitated by the optimal values of the pressing force and humidity of the raw mix selected during the study. They are important technological parameters. The resulting high-strength gypsum composite is characterized by a fine-crystalline structure with higher performance indicators than conventional gypsum materials due to the screening of the moisture effect on it.

Keywords: particle size distribution, packing density, optimization, computer modeling, structure, properties, calcium sulfate dihydrate

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1. INTRODUCTION

One of the most important problems of the building materials industry is the development of domestic production of efficient building materials based on harmonious and balanced activities in relation to the environment, saving material and fuel and energy resources, maximum use of local and man-made raw materials [1]. Such production should fully meet the UN Sustainable Development Goals and be bio-positive.

Safety issues in the conditions of high-tech human production activities are aimed at the use of building materials with reduced aggressiveness of impacts on the ecosystem as a whole and on humans in particular.

Gypsum-containing composites are exactly such materials. They ensure health and high quality of life in everyday conditions, reduce man-made impact in critical conditions. The absence of highly dispersed dust and toxic gas emissions that are dangerous to human life and high resistance in fire conditions distinguish them from other modern materials.

The use of energy-efficient non-firing production technologies along with the replacement of natural raw materials with industrial waste increases the demand and cost-effectiveness of gypsum products. The use of gypsum waste to produce products will not only reduce the cost of the resulting products, but also involve valuable man-made raw materials in production.

When considering and analyzing the evolutionary route of the formation of the solid state of matter, the main stages were identified – the origin of the phase, the growth of particles, their agglomeration, spontaneous and self-organized transformation in time, transitions between stages were designated, and the phenomena and processes of molecular (ionic), topological and morphological selection were discussed. These stages and phenomena, their processes should be considered as "objects" of nanomaterials impacts in order to achieve the effects of modifying the structures of building composites [2]. Developments in the field of nanomaterials are associated with the introduction of nanosized components into the primary raw material composition in order to improve the technological characteristics of the molding mixture and increase the physical and mechanical properties of the material. The effectiveness of using nanomodification can be achieved with the ability to control the mechanism of structure formation, the study of which will allow you to control the process and get the maximum effect from the minimum amount of the modifier used [3, 4].

Ultra- and nanodispersed additives have a significant effect on the hydration and crystallization of various binders, as well as on the physical and mechanical properties of composites [5-7]. The effect of a complex additive consisting of caustic magnesite and multi-walled carbon nanotubes (MWCNTs) on the structure and properties of a binder from natural anhydrite of the Ergachevskoye deposit was studied. In the course of the studies, based on a complex additive, a better result is achieved than with their separate use, probably due to the manifestation of a synergistic effect, respectively, the increase in compressive strength of anhydrite binder reaches 150 % with an optimal content of magnesite of 3% and 0.001% MWCNTs. A denser and stronger structure is formed, the additives act as crystallization centers and structure the anhydrite matrix, ultra- and nanoparticles contribute to the filling of pores of various sizes and the formation of a high-density structure. It is also known that MWCNTs with the addition of microsilica can be used to improve the properties of gypsum cement-pozzolanic binders, which improves their mechanical properties (the increase in strength at 28 days is 52 % compared to the control sample) and increases water resistance (by 35% compared to the control sample) due to the compaction of the composite structure [5, 6-8].

The structure of cement-sand mortar modified with basalt fiber and multi-walled carbon nanotubes was studied. In the course of the studies, the authors established that compacted structures, primarily of calcium hydroxide, are formed around the basalt fiber. The obtained micrographs indicate a decrease in shrinkage cracks, and, accordingly, a compaction of the structure of the cement paste. The optimal content of basalt fibers was 0.3-0.4 % of the cement mass. Basalt fiber made from rocks does not react with acids and water, but at present there are different points of view on the mechanism of destruction of basalt glasses by alkalis. The problem of leaching of basalt fiber in cement paste in the works [5, 7] is solved by structuring the cement paste on the surface of reinforcing basalt fibers with a dispersion of multi-walled carbon nanotubes.

The main specificity of the technology of nanomodification of building materials is the complexity of statistically uniform distribution of small doses of primary nanoparticles in them, which are prone to aggregation. At the heart of the mechanisms of action of nanoparticles, the author [8] noted the topological effect as one of the important surface effects with localization of nanoparticles in defects and ultramicrovoids of the dispersed system at the time of its formation. The author studied nanomodification in the field of building ceramics by introducing an aqueous solution of a grafted copolymer of acrylic polycarboxylate and polyethylene glycol stabilized with sodium chloride. In this case, a decrease in shrinkage of the raw material is observed, the average density increases, and the compressive strength increases by 30%.

It is known [9] that the adhesive strength of a gypsum binder in the presence of a mineral modifier in the form of potassium silicate cement increases due to the alkaline environment created in the gypsum paste and the activation of the interaction of calcium sulfates with water and due to the presence of silicic acid gel. The G-5 brand was used as the main binder. However, the authors found that the use of only potassium silicate cement as a modifying additive to a gypsum binder leads to a reduction in the setting time of gypsum by 2-2.5 times.

In the course of the studies, it was established that a modifier based on polycarboxylate has a significant plasticizing effect - a decrease in the viscosity of the cement paste by 48 %, which leads to compaction of the structure of the cement matrix in concrete, thereby contributing to an increase in the strength of cement concrete [10].

In their work [10], the authors study the effectiveness of modifying composites with nanosized barium hydrosilicates. However, the authors state that for composites with a non-uniform structure, such as cement and gypsum, the change in the property indices is not expressed for several reasons: the aggregative stability of the nanomodifier, the features of the chemical composition and surface structure of the nanoparticles, and the defective structure of the modified matrix. All this leads to a non-uniform distribution of nanomodifier particles, which leads to an increase in the density of the structure of certain areas of the material [10-12].

The work [13] studies the effect of highly dispersed iron-containing modifiers for anhydrite binders in order to improve their technological and physical-technical properties. The results obtained during the studies indicate an increase in strength and a decrease in water absorption of samples at the age of 28 days with an optimal content of additives, setting times are reduced, and the required mobility and cohesion are maintained. Such concretes on composite anhydrite binders can be used as floor compositions.

The authors of consider the issues of optimizing the size and morphology of gypsum binder particles, creating a high-density filler packing, which leads to the optimization of the microstructure of gypsum cement paste. In the work, a silica-containing technogenic raw material was used as an active mineral additive – concrete scrap, which was ground during the research to a specific surface of 500 m^2 / kg with subsequent mixing with Portland cement and gypsum binder. Dense particle packing, selected based on calculations [14], ensures the formation of a rigid framework in the concrete structure and allows optimizing the microstructure of gypsum cement paste, as well as increasing its strength by 24 %. According to the results obtained, the main cementing substance in the contact zone is calcium sulfate dihydrate and cement hydration products.

Cast gypsum concretes are known based on composite gypsum binder with the use of an active mineral additive. In their work, the authors varied two factors: the amount of composite gypsum binder and the amount of sand of fraction 5-2.5 mm. The results of the conducted studies showed that obtaining cast concrete on composite gypsum binder with its high density is possible with a content of coarse sand fraction of 50-70 % [14].

Currently, one of the new and promising areas of construction materials science is the study of the structure of dispersed systems. When creating materials at the stage of their development, it is necessary to conduct a large volume of expensive laboratory studies. In this regard, the study of the structure of dispersed systems necessitates the use of modern methods of mathematical modeling [15], the creation of computational models using computer technologies [16-19].

Currently, the industry has a number of technologies for the production of gypsum fiber products, where plant fiber uniformly distributed in the gypsum mass is used as reinforcement, for example, in the works, gypsum concrete composites reinforced with basalt fibers are presented [15]. Cellulose

fibers, chopped basalt fibers [15], glass fibers [17] and polypropylene fibers were used as reinforcement [18, 19]. However, tests in this work using basalt fiber did not show effectiveness.

The process of formation of the structure of gypsum stone obtained by the method of semi-dry pressing, the optimal grain composition with the use of a modifying additive is studied [20-25]. It is shown that optimization of the granulometric composition of powder mixtures with highly dispersed modifiers is impossible without the use of modern methods of mathematical modeling, creation of computational models using computer technologies. There is a need for the development of mathematical models. Optimization of the grain composition, the process of structure formation of unfired dispersed systems and technological modes – mathematical modeling allows a comprehensive description of the control of such systems, which is a relevant, modern and necessary task [15, 26].

High physical and mechanical characteristics of composites are ensured by the use of gypsum binders as the main raw material and ultra-dispersed modifier – waste from the production of basalt fiber [26].

The use of basalt dust waste as a modifier in the composition of building products for industrial and housing construction will improve the environmental component of production, as well as reduce the cost of products several times.

Various modifying additives have been studied in sufficient detail in the literature: caustic magnesite, carbon nanotubes, microsilica, potassium silicate cement, polycarboxylate, nanosized barium hydrosilicates, highly dispersed iron-containing modifiers, and silica-containing technogenic raw materials. The effects of each of these additives on the properties of binders are described in detail. The results and conclusions of the studies on adding the corresponding modifiers to the composition of certain binders are presented [16-19, 22-24]. It has been shown that the introduction of ultradispersed modifiers into the composition of pressed gypsum composite allows increasing its strength by 3 times [17, 21]. However, the introduction of modifiers that do not meet the physicochemical homogeneity with the original main substance can change the crystallization route [21, 22, 25, 27].

The results of the reviewed works made it possible to draw basic conclusions about the degree of study of modifying additives and their positive influence on the structure and properties of the binder – dihydrate technogenic gypsum [28].

Since this research work examines and studies the influence of dusty waste from the production of basalt fiber on technological modes, as well as on the structure and properties of unfired binders, in particular gypsum, the necessary set of properties in composite materials is achieved not by creating a new substance, but by successfully combining already known substances in one material, which was the purpose of this work.

To achieve the stated goal, the following tasks were solved:

• study the effect of pressing pressure on the properties of unfired gypsum composites modified with dust-like waste from the production of basalt fiber;

• study the effect of humidity on the properties of unfired gypsum composites modified with dust-like waste from the production of basalt fiber, using two-factor analysis;

• develop recipes for an effective unfired material reinforced with dust-like waste from the production of basalt fiber.

During the research, it was important to correctly select the sequence of solving the above-mentioned problems. Considering the relationship between them, it was necessary to establish whether there was a need to use the results of one of them when solving others. Based on the accepted working hypothesis, the sequence of completing the tasks was adopted as follows:

- study of the influence of pressing pressure on the properties of unfired gypsum composites modified with dust-like waste from the production of basalt fiber,

- study of the influence of humidity on the properties of unfired gypsum composites modified with dust-like waste from the production of basalt fiber.

Using two-factor analysis, the stages were combined. Then, the solution to the third problem was implemented – the development of stable gypsum binders with a reinforcing additive based on waste from the production of basalt fiber.

2. METHODS AND MATERIALS

In order to study the influence of quantitative characteristics of dust-like wastes from basalt fiber production on the main characteristics of an effective pressed gypsum composite, experimental compositions of mixtures of calcium sulfate dihydrate with basalt dust-like wastes were evaluated. Specific surface area (according to Blaine) of dust-like wastes from basalt fiber production is about 350 m^2/kg.

The studies of sustainable composites were carried out using as the initial material - dihydrate technogenic gypsum from used molds for casting building ceramics and faience of the Samara plant for the production of building ceramics and faience.

For the production of Peshelan gypsum binder for molds (Table 1, 2), high-quality gypsum stone of only grade 1 is used. According to the requirements of Russian Standard GOST 4013-2019 "Gypsum and gypsum-anhydrite stone for the production of binders. Technical conditions", its composition must contain at least 95 % calcium sulfate dihydrate. This allows classifying the resulting waste as high-quality secondary material resources due to their high chemical purity [16, 27, 28].

Table 1. Modification composition of gypsum binder β-modification.

Type of modification	Value, %
Semi-hydrate gypsum $CaSO_4 \cdot 0{,}5\ H_2O$	89.4
Soluble anhydrite $CaSO_4$	1.72
Unfired gypsum dihydrate $CaSO_4 \cdot 2\ H_2O$	1.3
Crystallization water	7.02

Table 2. Chemical composition of gypsum binder β-modification.

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	P_2O_5	F
0.8	traces	traces	–	37.52	0.10	53.78	0.05	0.007	–	–

The genesis of gypsum waste affects the structure of the raw material and the properties of the materials and products obtained on its basis [23, 24]. The waste of the Samara production has a large-crystalline structure with more perfect crystals of dihydrate technogenic gypsum compared to the waste of other industries. When using molding gypsum and the casting method of obtaining products (in this case, molds and models), favorable conditions are created for the recrystallization of dihydrate gypsum, the unification and enlargement of crystals in the free pore space.

It is known that a highly dispersed binder (Table 3) with high water demand is used to manufacture the molds at the plant. Therefore, the structure of dihydrate technogenic gypsum obtained on its basis is characterized by high porosity – 52% and, as a consequence, a large-crystalline structure.

Table 3. Main characteristics of Peshelan gypsum binder.

Binder grade	Specific surface area, cm^2/g;	Water absorption, %;	Volumetric expansion, %	Insoluble in HCl impurities, %
G - 6 B III	13580	40.4	0.13	0.17

In order to study the influence of quantitative characteristics of dust-like waste from basalt fiber production on the main characteristics of pressed gypsum composite, experimental compositions of mixtures of calcium sulfate dihydrate with basalt dust-like waste and the structures of hardened gypsum composites based on them were evaluated.

All studies of dispersion, granulometric, mineralogical and chemical composition were carried out using standardized methods and verified laboratory equipment. The specific surface of the powders of dihydrate technogenic gypsum was estimated by the filtration method by a PSH-11M device. The grain composition of the powders of dihydrate technogenic gypsum was estimated based on the results

of dispersion analysis using a laser analyzer of the FritschParticleSizer «Analysette 22» type.

The mineralogical composition of gypsum rock was assessed using powder X-ray diffractometry (XRD) with the use of an «ARL X'tra» diffractometer.

The microstructure of the starting materials and the resulting stable gypsum composite was evaluated using a JEOL JSM-6610LV scanning electron microscope in secondary and reflected electron modes at an accelerating voltage of 15 kEv. Sample preparation consisted of preparing chips and sputtering a conductive Pt layer. The JEOL JSM-6610LV (Japan) is a scanning electron microscope with a magnification of 5 to 300,000, an accelerating voltage of 0.3 kV to 30 kV, a resolution of 2.5 nm in high vacuum mode (30 kV) and 4 nm in low vacuum mode.

The experimental compositions of dry mixtures of calcium sulfate dihydrate with dusty waste from the production of basalt fiber were prepared by mixing in a laboratory mixer in accordance with the developed work plan. Mixing was carried out for 10 minutes. Then the mixtures were mixed with water and mixed for another 10 minutes until a homogeneous mixture was obtained.

The samples (Fig. 1) after semi-dry pressing using a hydraulic press (Fig. 2, 3) and a matrix (Fig. 1) for 7, 14 and 28 days hardened under normal dry conditions.

Fig. 1. Sample after pressing and mold for pressing.

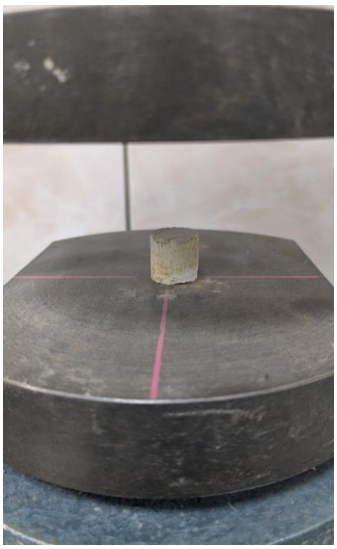

Fig. 2. Sample under hydraulic press testing.

Fig. 3. Laboratory hydraulic press with measuring scale.

The molding of cylindrical samples with a diameter and height of 50×50 mm, respectively, was carried out using hyperpressing.

In order to determine the average density values of gypsum stable composite samples, cylindrical samples were weighed and then kept at a constant temperature until a constant mass was reached in accordance with Russian Standard GOST 6428-2018.

3. RESULTS AND DISCUSSION

Man-made dihydrate gypsum, in its phase and chemical composition, unlike most man-made waste, is identical to natural gypsum rock, which is confirmed by chemical, X-ray diffraction and differential thermal analysis data. They are shown in Fig. 4, 5. The derivatogram (Fig. 4) is characteristic of dihydrate gypsum ($CaSO_4 \cdot 2H_2O$). When examining the DTA, DTG, TG curves, it is evident that in the temperature range of 80 – 125^{0}C a loss of adsorption water is observed. The most intense mass loss occurs in the temperature range of 140 – 240 ^{0}C, which is characterized by two endothermic peaks at 180 ^{0}C and 210 ^{0}C. The first effect corresponds to the removal of 1.5 water molecules from gypsum. The temperature of the first peak of the end effect is – 180 ^{0}C indicating a large-crystalline structure of the material. The second endothermic effect is less intense and is caused by the removal of the remaining 0.5 water molecules. The exothermic effect in the temperature range of 380 – 440 ^{0}C is associated with the inversion of $CaSO_4$, during which the crystal lattice is restructured. In the temperature range of 750 – 840 ^{0}C decomposition of $CaCO_3$ is observed (dissociation of 44 % CO_2). The nature of this endothermic effect and the loss of mass in the corresponding temperature range allow to assume that in the studied sample of dihydrate gypsum the amount of $CaCO_3$ does not exceed 2 – 3%.

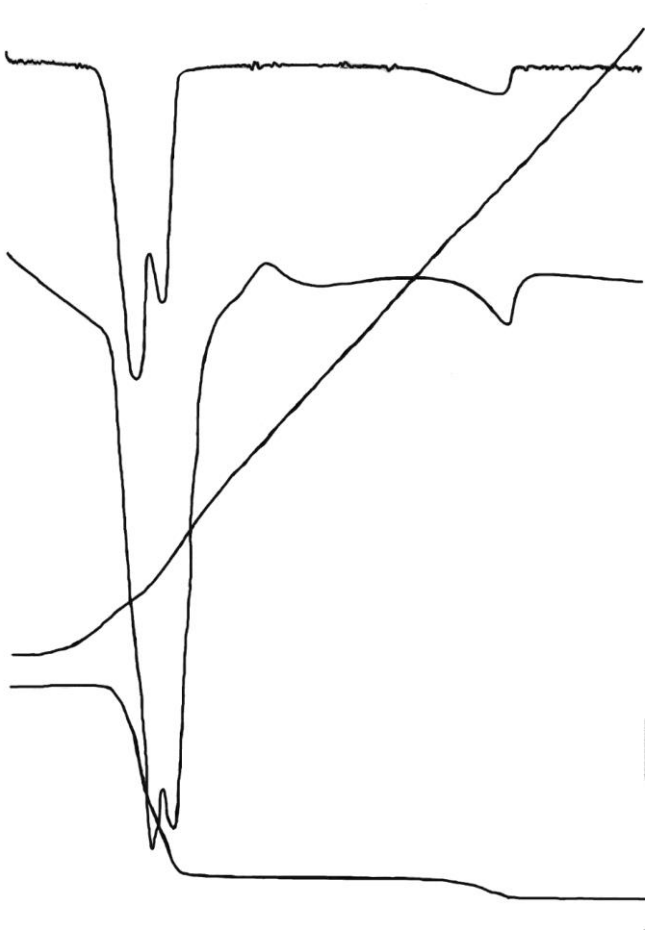

Fig. 4. Derivative diagram of dihydrate technogenic gypsum – waste from faience factory molds.

The XRD patterns of gypsum-containing waste, shown in Figure 5, confirm the data of derivatographic studies. The provided XRD pattern is characteristic of dihydrate gypsum (diffraction lines 7.661; 4.301; 3.818; 3.074; 2.885; 2.797; 2.69; 2.603; 2.501; 2.222; 2.144; 2.083; 1.998; 1.903; 1.81; 1.667 Å). Diffraction lines 3.50; 1.852 A indicate an insignificant content of anhydrite ($CaSO_4$). Impurities present in small quantities in the studied gypsum sample are presented in the form of feldspars – Ca $\{Al_2Si_2O_8\}$ (diffraction lines 3.187; 2.51; 2.135; 1.834 A) and pyrite – FeS_2 (lines 2.696; 2.411; 2.21 A). Diffraction reflections of calcite ($CaCO_3$), quartz (SiO_2), as well as clay minerals were not detected in the X-ray diffraction pattern.

According to the chemical analysis, this waste is of the first grade in terms of calcium sulfate dihydrate content and contains virtually no impurities; the content of $CaSO_4 \cdot 2\ H_2O$ in the waste is 98.54 %. The presence of calcium sulfate hemihydrate in the technogenic waste after its milling was not detected.

According to the results of radiation control (Russian Standard GOST 30108), gypsum has a low specific effective activity ERN and belongs to the first class, which guarantees its environmental safety when used in the construction industry [29].

Thus, the spent gypsum molds of the faience factory, in terms of their chemical composition, environmental friendliness and physical state, are a pure product obtained in a recyclable form and can be used directly in the technological process of obtaining gypsum materials and products.

In order to utilize high-quality waste with high chemical purity in the form of spent gypsum molds, an attempt was made to obtain a secondary gypsum binder based on it using a traditional firing scheme under plant conditions. The waste was crushed in a laboratory jaw crusher (Fig. 6) with subsequent firing with exhaust gases at a temperature of over 400^0C, after which the gypsum was ground in a laboratory activator mill (Fig. 7).

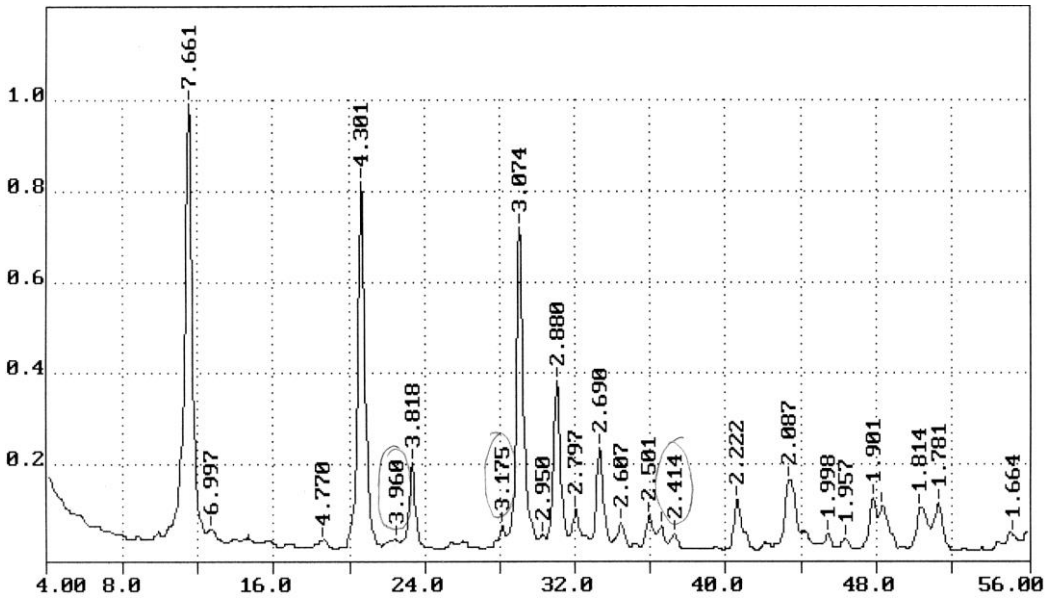

Fig. 5. X-ray di of dihydrate technogenic gypsum – waste from molds of the Konakovo faience factory.

The obtained secondary gypsum binder did not meet the consumer's requirements either in terms of its physical and mechanical characteristics (low strength and high-water demand), as shown in Table 4, or in terms of its economic efficiency, in connection with which the possibility of increasing its main physical and mechanical characteristics using additional technological methods was studied [30].

The *density* of pressed gypsum composites was determined by the formula:

$$P = m/V ,$$

where ρ_0 – density, kg/м^3, m – sample mass, kg, V – sample volume, m^3.

Total porosity was determined by the formula:

$$PHI = (1-\rho_0 / \rho)\times 100 ,$$

where PHI – total porosity of pressed gypsum composites, ρ_0 – average density, kg/m^3; ρ – true density of pressed gypsum composites, kg /m^3.

The *relative density* of pressed gypsum composites was determined by the formula:

$$d = \rho_0 / ,$$

where d – relative density of pressed gypsum composites, ρ_0 – average density of pressed gypsum composites, kg/m^3; ρ_0 – density of water, kg/m^3.

Specific strength R_{sp} was determined by the formula:

$$R_{sp} = R_{comp} / d,$$

where R_{sp} – specific strength of pressed gypsum composites, MPa; R_{comp} – compressive strength, MPa; d – relative density of pressed gypsum composites.

To improve the quality of the secondary gypsum binder of the faience plant, additives of lime and ash from the hydro-removal of Tverskaya TPP-4 were introduced. The introduction of peat ash up to 30% and a saturated lime solution for mixing gypsum increased the strength indicators of products obtained by casting technology based on the secondary fired gypsum binder of the faience plant, but the achieved strength of the obtained materials also did not meet the requirements of the consumer.

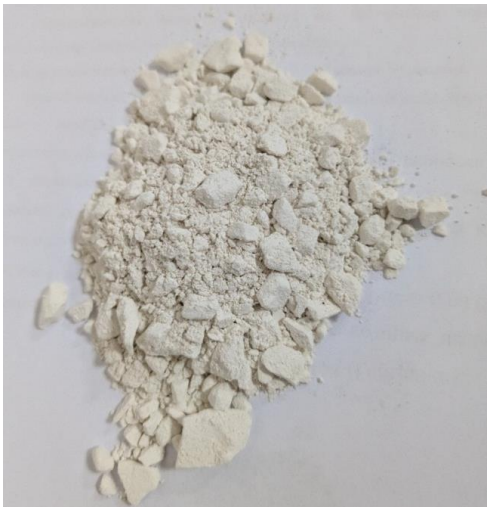

Fig. 6. Gypsum stone after fragmentation.

Fig. 7. Laboratory mill-activator.

Table 4. Comparative characteristics of physical and mechanical properties of primary gypsum binder and secondary gypsum binder obtained on the basis of dihydrate technogenic gypsum.

Name of indicators	Gypsum binder for faience factory	Secondary gypsum binder
Water requirement, %	40.4	80
Setting time, min.:		
initial	6	4.5
final	10.5	10.5
Compressive strength, MPa	6.26	1.58
Grinding fineness (residue on sieve No. 02), %	0.3	9

Binary raw material mixtures of calcium sulfate dihydrate were used in the form of a mixture of powders with different degrees of grinding (Fig. 8).

Fig. 8. Dihydrate technogenic gypsum after grinding.

The powders themselves were obtained by initial crushing of waste in a jaw crusher and subsequent grinding (Fig. 8). The grinding was carried out in a laboratory activator. Binary dry mixtures of technogenic gypsum (Fig. 9) were obtained by mixing them in a laboratory mixer. The ratio of powders of different degrees of grinding was taken as constant.

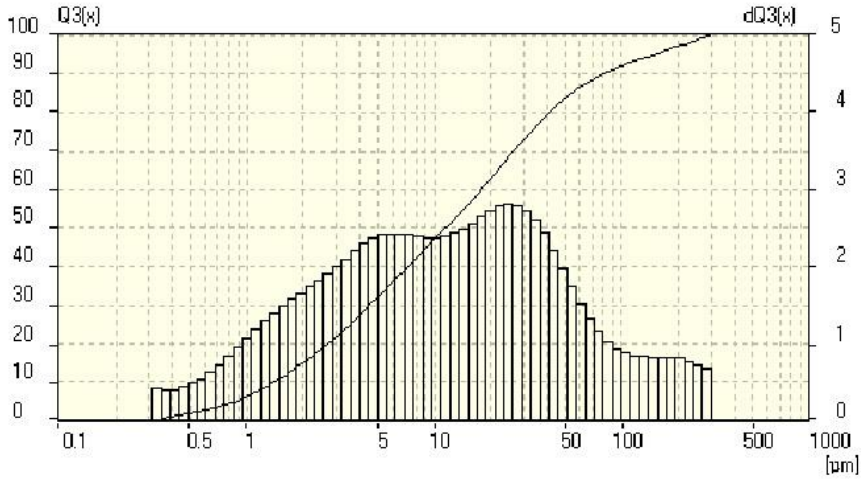

Fig. 9. Granulometric composition of a binary mixture of powders of dihydrate technogenic gypsum.

The mineral part of the compositions based on calcium sulfate dihydrate also included man-made basalt powder as a reinforcing component (Fig. 10-13). True powder density was 2182 kg/m^3.

The diffraction pattern of the powder sample is shown in Fig. 10, the results of the analysis of basalt powder samples for mineralogical and chemical composition are presented in Table. 5, 6.

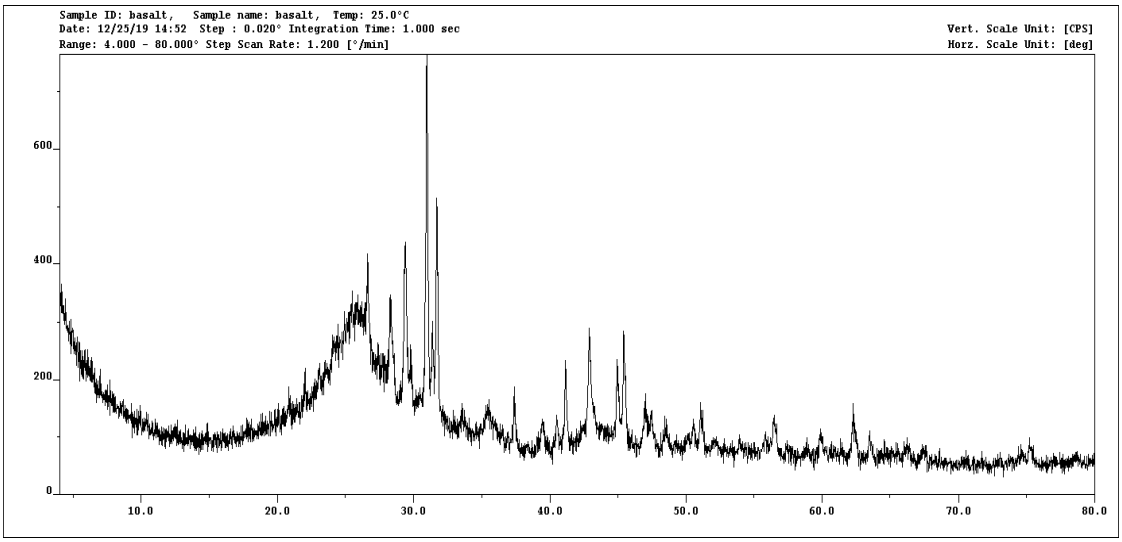

Fig. 10. Diffraction pattern of a sample of man-made basalt powder.

The studied samples of powdered waste of pulverized basalt are mainly represented by quartz, calcite, anorthite (Table 5). The chemical analysis showed that the pulverized basalt mainly contains silicon oxides - SiO_2, (41.13 %), calcium - CaO (14.66 %) and magnesium - MgO (13.81 %). Also present are oxides of iron Fe_2O_3 (7.34 %), sodium Na_2O (6.59 %), aluminum Al_2O_3 (3.99 %) and potassium K_2O (4.14 %). Zinc oxide and sulfur are contained in an amount slightly more than one percent - 1.57 and 1.11 %, respectively. The chlorine content is 4.29 %.

Table 5. Mineralogical composition of man-made basalt powder.

Quartz	Calcite	Dolomite	Periclase	Sodium chloride	Potassium chloride	Anhydrite	Amorphous phase
5.1	7.6	19	5.9	6.1	1.8	5.0	50

The large mass of basalt powder is glassy, permeated with numerous crystals of plagioclase, pyroxene (Fig. 11, 12). Studies of the composition of the sample also established the presence of 60 % amorphous component (Fig. 10).

Table 6. Chemical composition of man-made basalt powder.

Compound	Wt %	Est.Error	Element	Wt %	Est.Error
SiO2	41.13	0.25	Si	19.23	0.12
MgO	14.66	0.18	Mg	8.84	0.11
CaO	13.81	0.17	Ca	9.88	0.12
Fe2O3	7.34	0.13	Fe	5.14	0.09
Na2O	6.59	0.12	Na	4.89	0.09
Cl	4.29	0.10	Cl	4.29	0.10
K2O	4.14	0.10	K	3.43	0.08
Al2O3	3.99	0.10	Al	2.11	0.05
S	1.57	0.06	S	1.57	0.06
ZnO	1.11	0.05	Zn	0.894	0.04
MnO	0.428	0.021	Mn	0.332	0.017
F	0.320	0.13	F	0.320	0.13
TiO2	0.262	0.013	Ti	0.157	0.0079
P	0.191		P	0.191	0.0096
CuO	0.0904		Cu	0.0722	0.0036
SrO	0.0323		Sr	0.0273	0.0014
Cr2O3	0.0207		Cr	0.0142	0.0008
Co3O4	0.0117		Co	0.0086	0.0009
NiO	0.0106		Ni	0.0084	0.0010

Fig. 11. Appearance of man-made basalt powder.

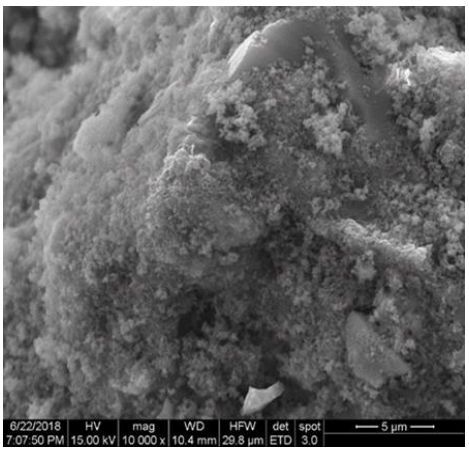

Fig. 12. Microstructure of man-made basalt powder.

SEM images of man-made basalt powder show that its isometric micro- and nanoparticles have a defective structure (Fig. 13). The volumetric coefficient, which takes into account the irregularity of the particle shape at different scale levels, averaged 0.428.

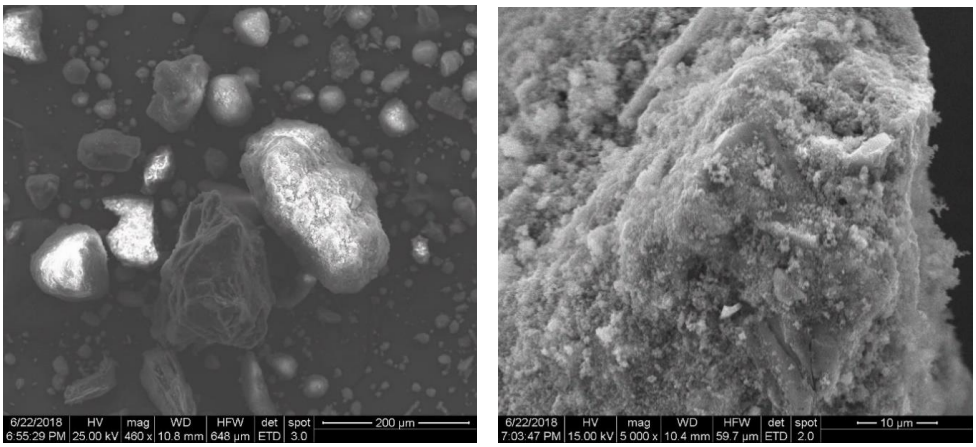

Fig. 13. Isometric micro- and nanoparticles of dispersed basalt powder.

Studies of the strength of pressed stone with a basalt modifier have shown that the introduction of dust waste has a positive effect on the compressive strength in the range of its content from 0 to 10 percent by weight (Fig.14).

A further increase in the quantitative content above 10 % has a negative effect on the strength. The drop in strength was more than 20%. The maximum strength with dust waste was about 40 MPa at a density of 1920 kg/m^3 at the age of 14 days of hardening (Fig. 14, 15). The kinetics of the change in the average density in the process of structure formation for compositions with varying percentage content of basalt dust are shown in Fig. 15. With increasing additive content, the density increases in the range of its change from 0 to 15% for samples of 7 and 14 days of hardening. At the age of 28 days, the nature of the dependence changes. At the age of 28 days, the effect of the additive is practically not manifested. The change in density at this age does not exceed 1.5%. All changes in density in this study did not exceed 2.5%.

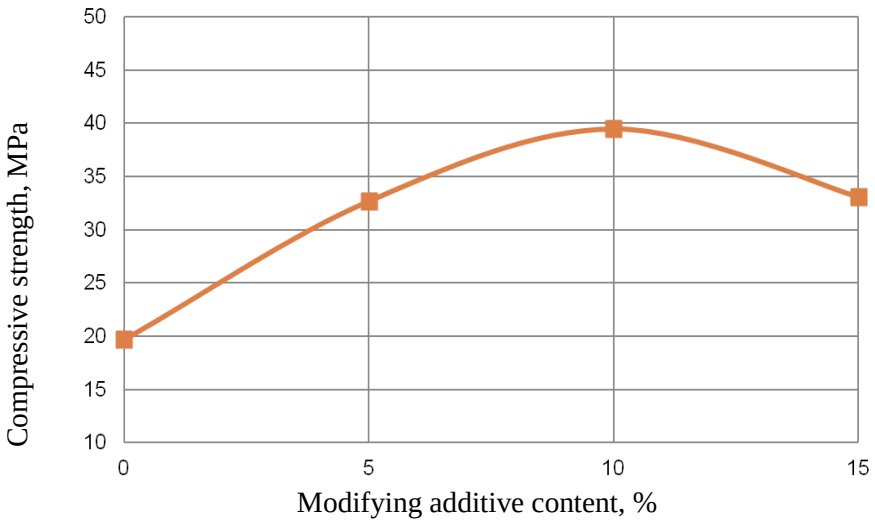

Fig. 14. Dependence of the strength of pressed gypsum composites on the 14th day of hardening on the percentage content of the reinforcing additive.

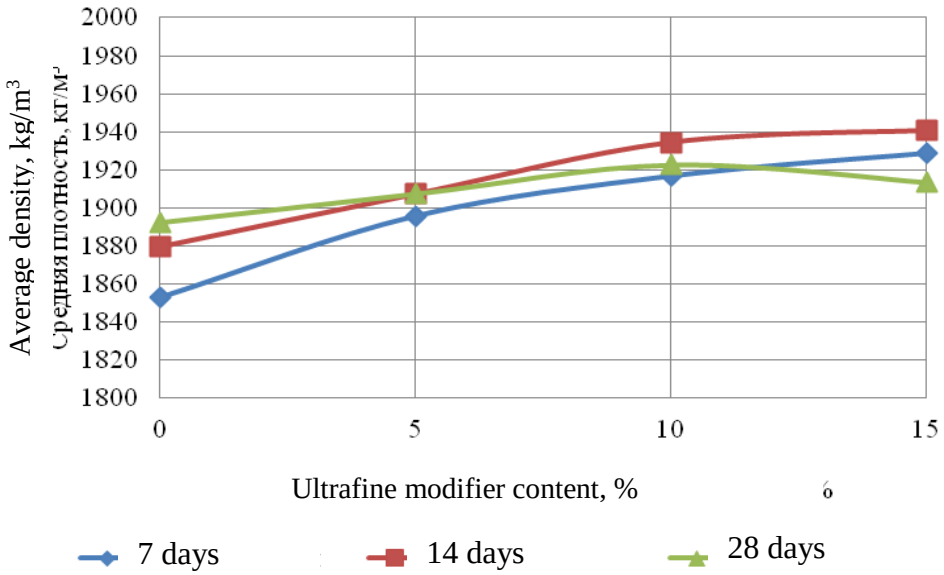

Fig. 15. Dependence of the average density of pressed gypsum composites on 7, 14 and 28 days of hardening on the percentage content of the reinforcing additive.

Electron microscopic studies of the structure of the obtained pressed samples of the stable composite are presented in Fig.16.

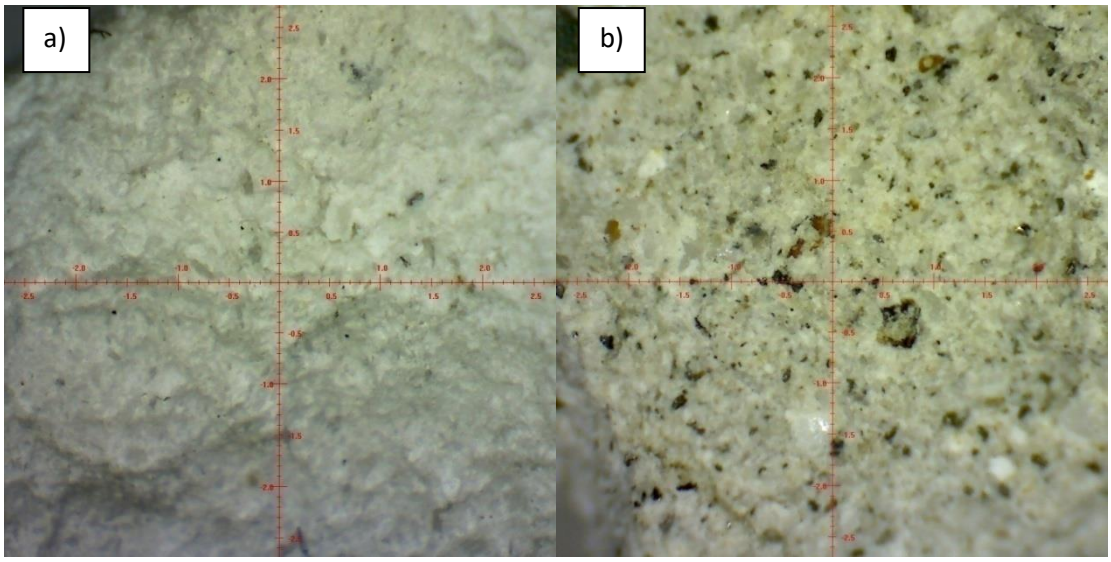

Fig. 16. Microstructure of pressed composite based on calcium sulfate dihydrate of standardized grain composition after 7 days of hardening: a) without additives; b) with crushing waste.

The analysis of the results showed that the optimal value of the basalt dust content in the composition of the pressed stable composite based on dihydrate technogenic gypsum is 10 %. This value was adopted as constant in further studies.

In order to study the influence of water demand and pressing force on the structure and properties of the unfired composite with basalt additive, a mathematical planned experiment was carried out, the results of which are presented in the data in Table 7, 8 and in Fig. 17-22.

Table 7. Strength values of stable composites for compositions 1 – 9, 28 days of hardening.

№ comp	W/S	Pressing pressure, MPa	Compressive strength, MPa	Specific strength, MPa
1	0.05	30	18.9	8.69
2	0.05	60	49.2	22.7
3	0.05	90	48.4	22.5
4	0.06	30	31.6	14.62
5	0.06	60	59.2	27.92
6	0.06	90	55.6	26.2
7	0.07	30	28.7	13.3
8	0.07	60	53.5	25.3
9	0.07	90	47.1	22.31

It was found that the pressing pressure affects the water requirement of the raw mix in accordance with Fig. 17, 19. The maximum average density of the composite is achieved with a minimum water content (W/S= 0.05) and pressing pressure (30 MPa), it was 2174 kg/m^3. An increase in the pressing force negatively affects the average density. A change in the water content (W/S ratio) has virtually no effect on the average density when it changes in the range from 0.06 to 0.07. The changes do not exceed the statistical scatter values.

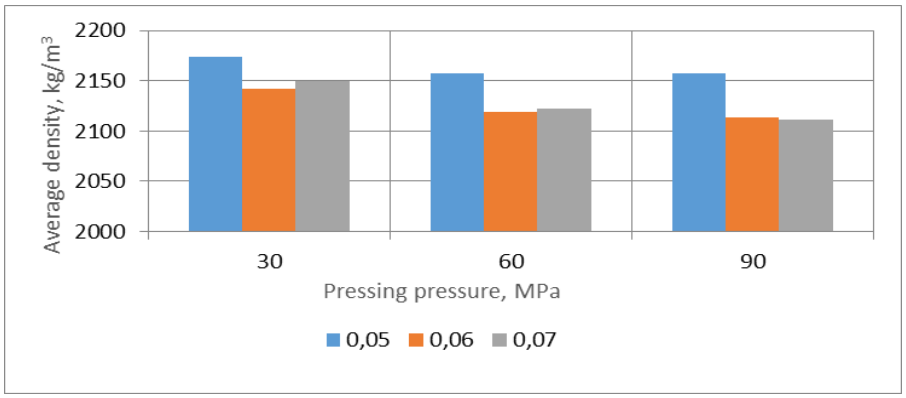

Fig. 17. Density diagrams of gypsum samples obtained at pressures of 30, 60 and 90 MPa (28 days of hardening).

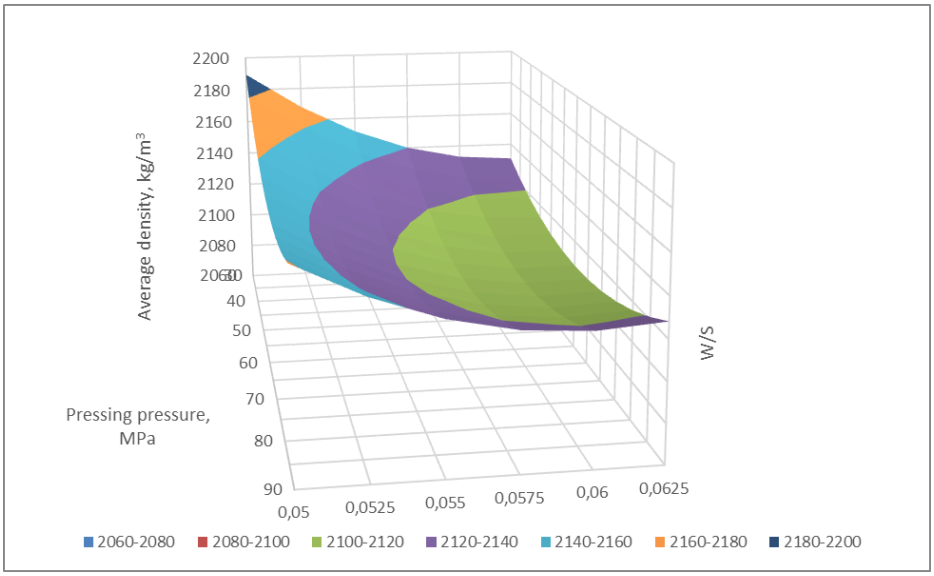

Fig. 18. Dependence of the density of pressed gypsum samples (28 days of hardening) on the pressing pressure and W/S.

The compressive strength increases in the case of water content W/S= 0.05 with an increase in pressure from 30 to 60 MPa – 68 % ((Fig. 19-20). However, a further increase in pressure leads to a decrease in strength, but a small one – the decrease in strength was 9 %.

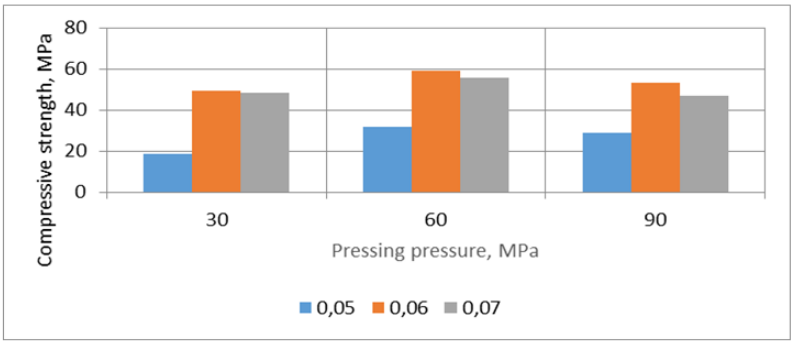

Fig. 19. Strength diagrams of gypsum samples obtained under pressure of 30, 60 and 90 MPa, (28 days of hardening).

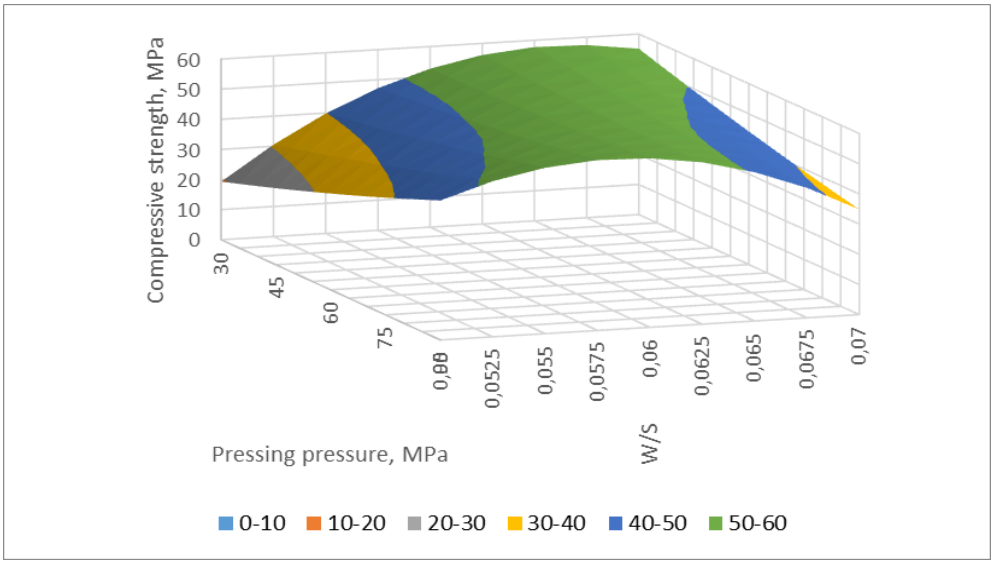

Fig. 20. Dependence of the strength of pressed gypsum samples (28 days of hardening) on the pressing pressure.

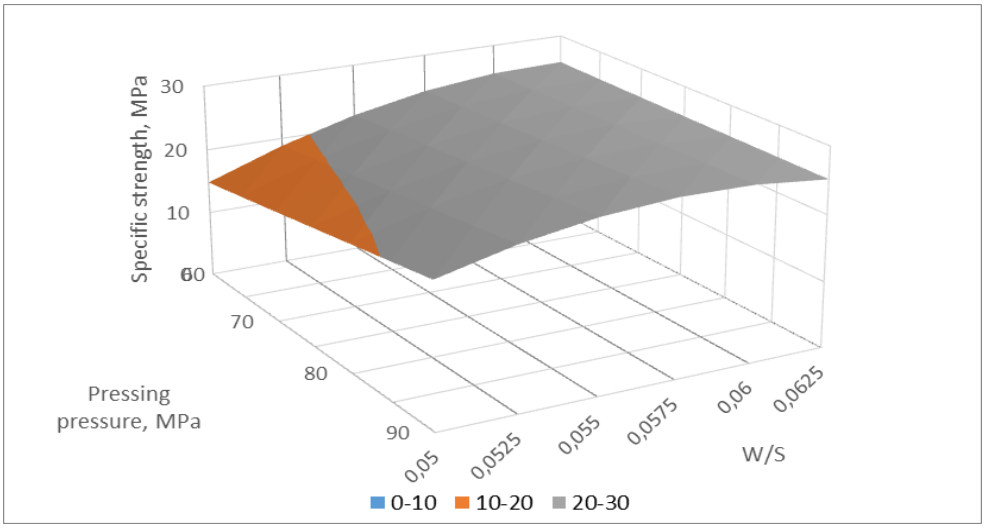

Fig. 21. Dependence of the specific strength of pressed gypsum samples (28 days of hardening) on the pressing pressure.

Table 8. Values of average density and total porosity of stable composite samples for compositions 1 – 9.

№ comp	Density, kg/m^3	Porosity, %
1	2174	1.2
2	2142	2.7
3	2150	2.3
4	2157	2
5	2119	3.7
6	2122	3.6
7	2157	2
8	2114	3.9
9	2111	4.1

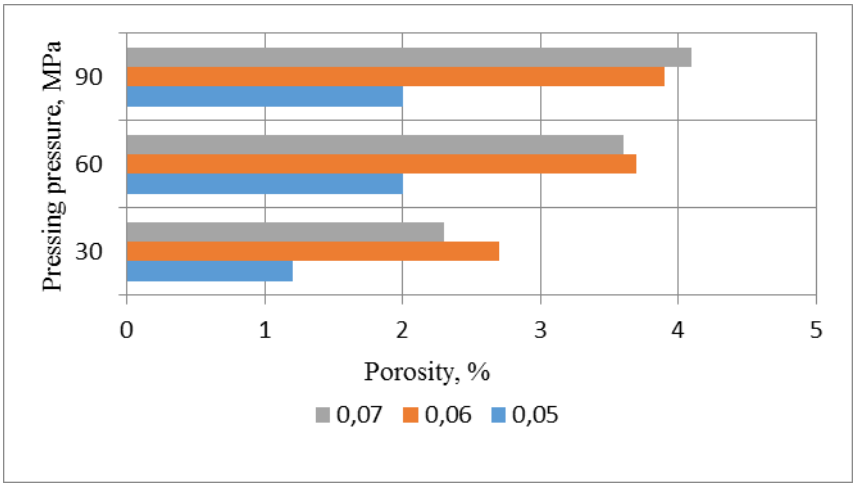

Fig. 22. Dependence of porosity of pressed gypsum samples (28 days of hardening) on pressing pressure at humidity: 5 %, 6 %, 7 %.

Since it is known that the hydration products of aluminates and aluminoferrites from the impurities of the used industrial additive give a clearly expressed picture of condensation-crystallization structure formation [20, 31], their inclusion in the processes of structure formation of unfired gypsum stone undoubtedly affects its properties. The maximum strength of the composite is achieved at a pressing pressure of 60 MPa and an optimal water content of 0.06 (Table 7), but the increase in strength compared to the composite obtained at a pressure of 30 MPa is only about 17 %, while energy costs increase. The highest strength values are achieved in the range of water content of 0.055 - 0.0675 depending on the pressing force (Fig. 20). A decrease in strength and average density when using a pressure of 90 MPa is explained by the decompaction of the mixtures.

Microstructural analysis showed that the structure of unfired gypsum stone of the control composition (without the use of basalt dust) is represented by large well-crystallized crystals of dihydrate, fairly uniform in size and habit (Fig. 23). Whereas in the structure of gypsum with the addition of a modifier there are polydisperse particles with the presence of a poorly crystallized phase (Fig. 24) and crystals close to "round shape"**.** It has been shown that in the presence of the additive, a strong fine-crystalline structure can be formed with additional filling of the pore structure with the most dispersed basalt particles (Fig. 13).

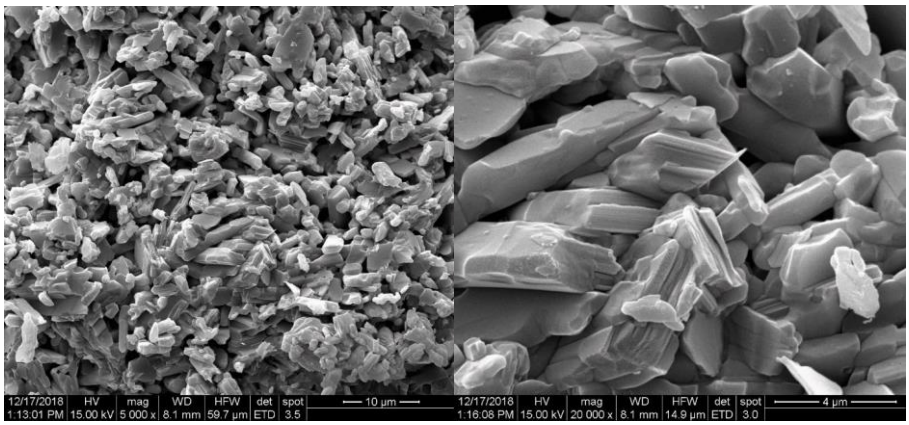

Fig. 23. Microstructure of unfired gypsum stone without additives (control composition).

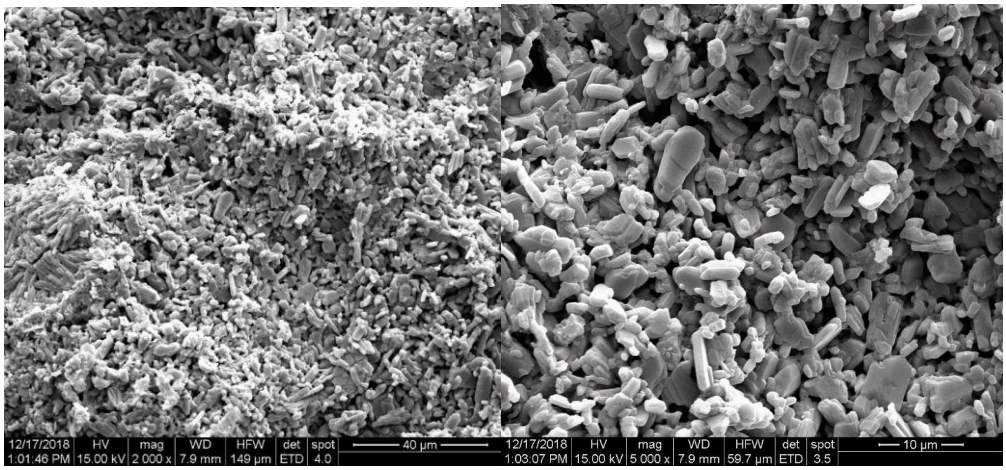

Fig. 24. Microstructure of modified gypsum unfired stone.

4. CONCLUSIONS

As a result of the analysis of the obtained data, the following conclusions can be drawn:

(1) It is possible to obtain stable composites based on gypsum stone waste and a reinforcing basalt additive, which is formed as a waste from dust removal in mineral wool production. The use of industrial waste and the method for producing gypsum stone products meets modern requirements in the field of sustainable development with an emphasis on resource conservation and reducing the carbon footprint in the production of mineral binders [31-34].

(2) The additive is characterized by a high degree of dispersion. This has a positive effect on the properties of the resulting composites. The optimal content of the additive from dust removal waste from basalt production is 10 %. The maximum strength of the unfired gypsum composite at the age of 14 days is 40 MPa.

(3) The patterns of change in the properties of the gypsum composite from the pressing pressure and water content in the raw mix were studied. The dependence of the strength of the samples on the pressing pressure showed an increase in strength with an increase in pressing pressure to 60 MPa.

(4) Water content has a significant effect on the strength and density of the composite. The identification and maintenance of optimal water content should be deeply studied as a technological parameter in the production of gypsum pressed products for bio-positive construction.

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